Environmental Aging and Deadhesion of Siloxane-Polyimide-Epoxy Adhesive

Steven Murray, Craig Hillman, and Michael Pecht, *Fellow, IEEE*

***Abstract*—Adhesives are subject to environmental aging that degrades their strength and fracture toughness over time. Aging increases the rate of crack growth under fatigue loading, and can lead to deadhesion of an adhesive bond. In this study, an empirical model is constructed from peel test data that quantifies the rate of aging of a siloxane-polyimide-epoxy adhesive as a function of temperature and humidity exposure. An accelerated test methodology that accounts for both aging and fatigue is then proposed, and demonstrated using a case study.**

***Index Terms*—Accelerated testing, adhesives, delamination, polymer aging.**

I. Introduction

ADHESIVES are susceptible to environmental aging that degrades their material properties over time [1]–[6]. Thermo-oxidative degradation and hydrolysis of adhesives can cause chain scission [4] and increased crosslinking [2], resulting in decreased strength and embrittlement. Metal substrates catalyze the aging of many adhesives bonded to them, such that the part of an adhesive joint contacting a metal substrate may degrade faster than that in the bulk of the material [6]. With aging, fracture toughness decreases and crack growth rate for a given fatigue loading increases [2], [7]. Aging can accelerate the rate of fatigue crack growth caused by thermo-mechanical and hygroscopic stresses resulting from changes in the temperature and humidity of the local environment, and lead to failure of an adhesive joint [1], [8]–[12].

Chemical degradation of adhesives is often analyzed by measuring percent weight loss as a function of time during aging at multiple temperatures [3], [4], and then modeled using an Arrhenius relationship. This approach has a number of weaknesses. Only degradation processes that uniquely result in mass being lost to the ambient environment are included. The moisture content must be kept constant throughout the experiment so that moisture absorption/desorption does not confound the results. Moisture concentration, which affects the rate of hydrolysis of adhesives, is not included as a variable in the degradation model. Chemical degradation is also not always directly correlated to mechanical properties, such as adhesion strength [2].

The metric used here to track the degradation in mechanical strength of adhesives with aging is peel strength. Test samples are aged at a variety of temperature and humidity conditions, their degradation in peel strength over time measured, and an empirical model for the degradation constructed using regression techniques. This method has the advantage of measuring the degradation in mechanical strength directly. If one of the adherands is rigid, or can be adhered to a rigid substrate, a 90° or 180° test can be used. If both adherands are flexible, a T-peel test can be used. Peel strength is expressed in terms of unit force per unit width of the strip being peeled.

Tight control over test parameters must be maintained in order for peel tests to be repeatable. Peel strength is a function of peel rate, peel angle, temperature, moisture content, metallization thickness, and metallization width [13]. Peel tests also create deformation within the bulk of the adherands, such that the force measured is not only the basic adhesion strength due to van-der-Waals forces, charge transfer interactions, covalent bonds, and mechanical anchoring, but also contains some error due to force expended deforming the adherands. The percentage of peel strength that is comprised of adherand deformation is proportional to the thickness of the adherands. If the samples can not be designed to avoid excessive plastic deformation of the adherands, or if the adhesion strength of the adhesive is very small, analysis may be required to estimate the energy expended in deforming the samples, such that this can be subtracted out and an estimate of fundamental adhesion strength made.

In this study, the aging of a siloxane-polyimide-epoxy (SPIE) adhesive on Cu-metallized substrates was characterized using T-peel tests, and an empirical model for the aging as a function of temperature and humidity developed. Thin adherands were used, such that the energy expended deforming the adherands was minimal, and the peel strength measurements could be applied directly. A test methodology that applies this model to the design of accelerated tests for adhesive qualification is proposed. This test methodology is then illustrated with a case study by designing a custom qualification test for a multi-chip module constructed with SPIE lamination adhesive.

II. Development of Aging Model

OxySIM 600, an adhesive designed for use as an overlay adhesive with polyimide dielectric and manufactured by Occidental Chemical Corporation, was aged at six different environmental conditions. The degradation in its peel strength with aging at each condition was tracked. Results of these studies were then used to create an empirical model for the degradation behavior as a function of temperature and humidity exposure.

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The authors are with the CALCE Electronic Products and Systems Center, University of Maryland, College Park, MD 20742 USA.

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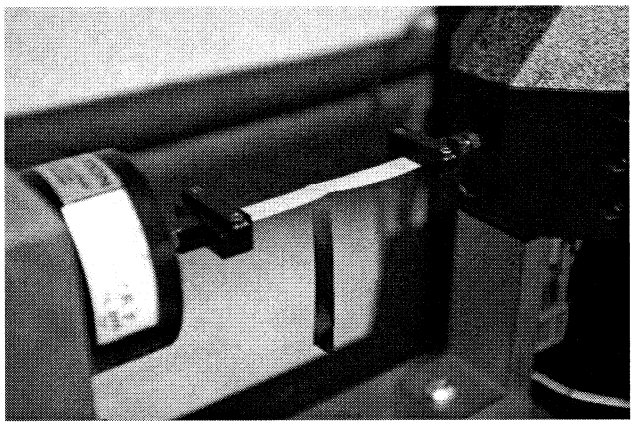

Fig. 1. T-peel test for evaluation of adhesive strength.

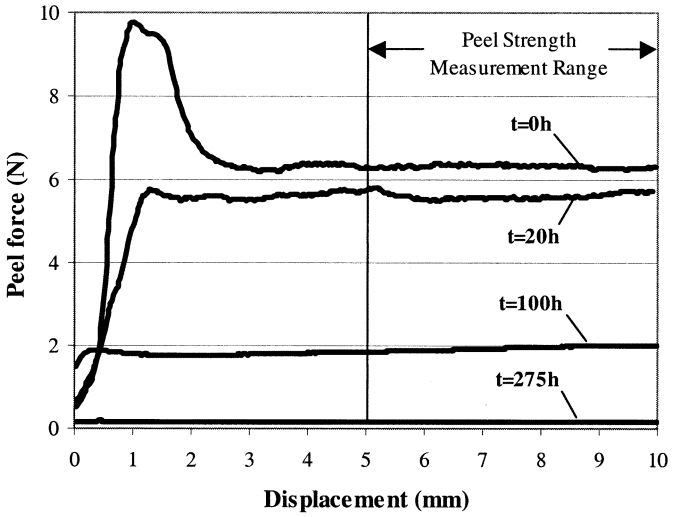

Fig. 2. Peel strength test results for a sample aged at 130 °C/85%RH.

A. Test Plan

Adherands were created by depositing 120 A of Ti, followed by 28 μm of Cu, on two plasma treated, 25.4 μm thick polyimide substrates. Spun-on SPIE of 12 μm thickness was used to adhere the Cu-metallized sides of the adherands together. Strips of 1.3 cm × 12.7 cm size were cut out for use as peel test samples.

The peel strength of each sample was measured using a T-peel test based on ASTM D1876 [14]. Prior to each test, samples were left out on racks in the laboratory for 24 hours to come to an equilibrium moisture content. Samples were then pulled apart at an angle of 180° at a constant rate of 2 cm/min for a distance of 1 cm in a MTS Tytron tensile tester. Due to the geometry of the samples, this rate results in 1 cm/min of adhesive bond separation. The bottom of the sample was left dangling such that it could be pulled up freely as the test progressed. The peel test is illustrated in Fig. 1. To calculate the peel strength for each sample, 150 sequential readings were taken by the data acquisition system during the final 0.5 cm of peeling and averaged.

Five coupons were then aged at each of 6 environmental conditions (85 °C/1%RH, 130 °C/1%RH, 150 °C/1%RH, 85 °C/85%RH, 130 °C/85%RH, and 150 °C/85%RH) using temperature/humidity and HAST chambers. Aging conditions were limited to 150 °C to keep below the glass transition temperature of the material (165 °C). At 0, 20, 100, and 275 h of aging, the samples were removed from the chambers and the peel test repeated on each sample. The percent degradation in peel strength from the baseline value for each sample was recorded. The results from the five samples at each aging condition were averaged together to give an average normalized peel strength value at each point in time.

B. Results

Peel strength degraded during aging, with both temperature and humidity affecting the rate of degradation. Sample force versus displacement curves are provided in Fig. 2. Failure occurred cohesively, but with an irregular and unstable fracture surface, leading to a fair amount of scatter in the data.

When the natural logarithm of peel strength was graphed vs. time for each aging condition, straight lines were obtained. Peel strength was therefore modeled using an exponential decay equation

$$P = 100 \cdot e^{-kt} \tag{1}$$

where P is the normalized peel strength, k is the reaction rate at the given level of temperature and humidity, and t is the aging time in hours.

The strength of the adhesive bond degrades due to hydrolysis and thermo-oxidative degradation of the adhesive, and possibly due to corrosion of the underlying metallization. The rate of corrosion and hydrolysis would be expected to be dependent upon humidity by a power relation and dependent upon temperature by an Arrhenius relation. The rate of thermo-oxidative degradation would be expected to be independent of humidity and dependent upon temperature by an Arrhenius relation. The overall reaction rate can be expressed as

$$k = A \cdot \mathrm{RH}^n \cdot e^{B/T} + C \cdot e^{D/T} \tag{2}$$

where RH is the relative humidity expressed as a decimal between 0–1, T is the absolute temperature in K, and A, B, C, D, and n are constants. If the reaction is modeled as first-order with respect to humidity, this equation reduces to

$$k = A \cdot \mathrm{RH} \cdot e^{B/T} + C \cdot e^{D/T} \tag{3}$$

when n is set equal to 1.

Non-linear regression was used to solve for the coefficients in (3) using the normalized peel strength vs. time data. The resulting equation was

$$P = 100 \cdot e^{-kt} \tag{4a}$$

$$k = 9290 \cdot \mathrm{RH} \cdot e^{-5460/T} + \left(2.44 \times 10^{10}\right) \cdot e^{-13\,000/T} \tag{4b}$$

where P is the normalized peel strength. Analysis of variance (ANOVA) found that the model fit the data with $R^2 = 0.95$. Graphs of the model overlaid on the original data are provided in Fig. 3.

The aging of the adhesive will significantly affect its reliability for virtually all practical application environments. The

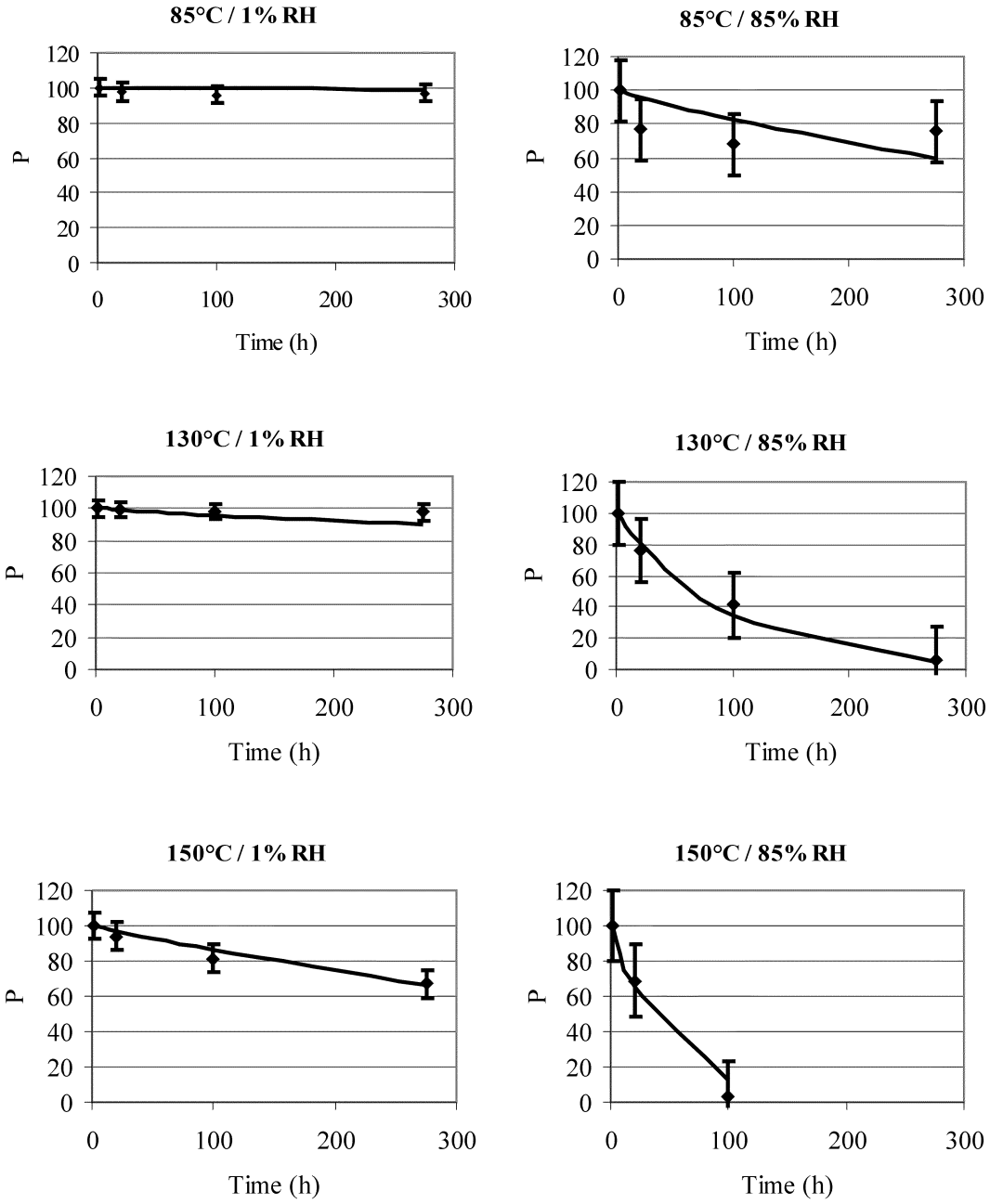

Fig. 3. Fit of regression equation to experimental data. Error bars represent ±1 standard deviation.

degradation of the adhesive strength predicted by (4) for various application conditions is illustrated in Fig. 4. This equation can be used to predict the decrease in resistance of the adhesive bond to crack propagation through the bulk of the SPIE during aging.

III. Qualification of Adhesives

The aging model developed in the previous section, (4), can be directly applied to accelerated test design. In this section, a brief review of the failure mechanisms of adhesives is provided. A methodology that takes aging into account when designing fatigue qualification tests for electronic products in then proposed.

A. Failure of Adhesives

Failure by separation of the material in a stackup can occur interlaminarly, intralaminarly, or with a mixed fracture surface consisting of a combination of the two. Interlaminar failures are sometimes called adhesive failures or weak interfaces, and intralaminar failures are sometimes called cohesive failures or strong interfaces [8]. Interfacial strength of a structural adhesive joint is usually stronger than the cohesive strength within the adhesive, such that failure occurs cohesively, but often in the near-interface region [13], [15], [16]. If a strong interface is obtained through proper pre-treatment and processing, the limiting factor is the strength of the adhesive itself. Surface roughness which provides mechanical anchoring,

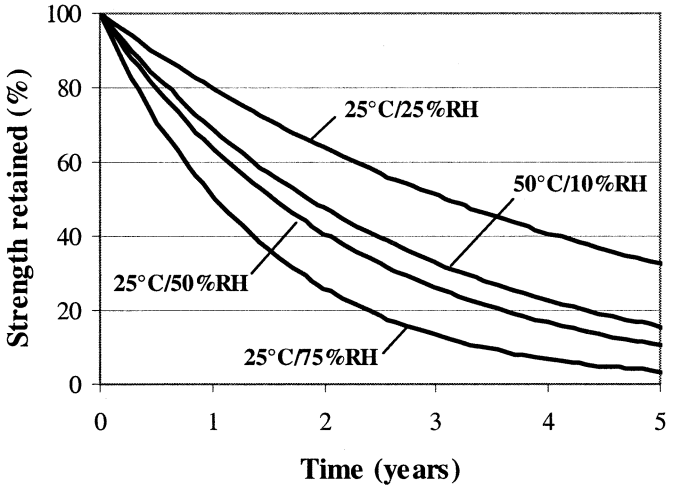

Fig. 4. Predicted degradation of SPIE at various application conditions.

the cleanliness of surfaces during processing, and the type of chemical bonds formed between the two materials all can affect interfacial strength. Silane coupling agents, which form chemical bonds between organic molecules in adhesives and inorganic substrates, are sometimes used to improve interfacial adhesion strength when bonding to inorganic materials [17], [18]. Epoxy joints are generally considered to be weaker under peeling or cleavage stresses than they are under pure shear or tensile loading [15].

The strength of adhesives with hygroscopic functional groups is a strong function of humidity. For these adhesives, such as acrylic or epoxy-amide, peel strengths at high humidity can be double that at low humidity [19]. Absorbed water generally has a plasticizing effect on adhesives, lowering its modulus and strength, with a linear dependence of joint strength upon water content [20]. Fracture toughness of polymers is a function of humidity as well [12]. Water concentration is also a primary factor in adhesive delamination [1]. The presence of water leads to lower bond stability, and applied stress decreases the bond potential and can lead to creep or fatigue [1]. Water can also disrupt secondary bonds across interfaces due to polar nature of water molecules, with water displacing the adhesive at an interface.

Ambient temperature can also affect the strength of an adhesive. Temperature affects properties because molecules become further apart as temperature increases, which decreases intermolecular forces holding molecules together. The material time constant is reduced with increasing temperature. Fracture toughness of polymers can also be a function of temperature [12], [21].

With proper processing, strong interfaces are formed when SPIE is adhered to Cu with Ti barrier metallization. Failure of the SPIE samples during peel testing occurred cohesively in this experiment. The failure mechanism of concern is therefore the propagation of a fatigue crack through the bulk of the SPIE adhesive.

B. Fatigue Crack Propagation

Stresses are the driving forces for fatigue crack growth that can lead to failure of an adhesive joint. The primary stresses within adhesives are usually thermo-mechanical and hygroscopic stresses resulting from changes in the local environment, or from power cycling of nearby electronic components. In some applications, mechanically applied loads such as bending or vibration may contribute to material fatigue as well.

Thermo-mechanical stresses arise due to coefficient of thermal expansion (CTE) mismatch between adjacent layers [8], [10], [11]. Both shear and normal ("peeling") stresses are generated. Stresses increase exponentially as distance from the neutral axis increases, with the largest stresses generally found near edges [11]. However, the presence of vias in the structure can greatly complicate stress fields, making them harder to predict analytically.

Hygroscopic stresses result from moisture absorption and desorption from the environment [1], [9]. Swelling due to moisture absorption is linearly related to the relative humidity of the surrounding air, where the proportionality constant is the coefficient of hygroscopic expansion (CHE). As moisture enters the polymer parts of the material stackup, compressive normal stresses build up around the edges where the moisture concentration is higher, balanced by tensile normal stresses in the center. At the same time, swelling induces shear stresses, with a maximum value approximately a quarter of the distance into the material [1]. Stresses resulting from moisture gradients decay as moisture equilibrium is reached, leaving only stresses due to global CHE mismatch with the substrates to which the adhesive is bonded.

Fatigue damage models are available for some adhesives based on crack growth rate measurements taken during fatigue testing of specially designed samples [22]–[26]. Simple models use a Paris law crack growth relationship. More sophisticated models use a summation of a Paris law term with a thermally activated term to account for stress-assisted hydrolysis of the bonds at the crack tip, similar to models for subcritical crack growth due to stress corrosion cracking in bulk glass. Crack growth rates are sensitive to temperature, humidity, stress cycle size, loading rate, and the mechanical phase angle of the loading.

C. Qualification Methods Used by Electronic Product Manufacturers

Reliability tests run by electronic product manufacturers are generally taken from industry or military standards. For example, the Institute for Interconnecting and Packaging Electronic Circuits (IPC), a leading standards authority for the printed circuit board industry [27], has several standards that detail tests that can be conducted to evaluate the quality and reliability of a variety of product types [28]–[32]. Such test plans generally specify a temperature cycling test with a predefined range and number of cycles, and sometimes specify a separate high temperature, high humidity aging test. Although such tests may be useful for supplier benchmarking and quality control purposes, they are inadequate for the qualification of products against material deadhesion. Shortcomings of such test plans include the following.

1) Effect of aging on fatigue resistance is neglected, as aging is not incorporated into the thermal cycling test, but is run separately, if at all.

2) No correlation exists between the harshness and duration of the aging test and the application environment in which the product will be used.
3) No correlation exists between the size and number of thermal cycles and the cycling that will be experienced in the application environment.
4) Hygroscopic stresses due to humidity cycling in the application environment are neglected (for nonhermetic product).

A better approach to qualification is to tailor qualification tests based on the physics of failure and the application environment of the product. Five major contributing factors to deadhesion of adhesives are materials, product dimensions, manufacturing processing, aging, and applied stresses. Since it is assumed that the materials, dimensions, and manufacturing processes used for the test samples are the same as those for the actual product, the two factors of primary importance for accelerated test design are aging and applied stresses. These factors in turn share the common sources of temperature and humidity. Oxygen concentration is assumed to be the same in all cases (standard atmospheric concentration). If present in the application, bending and vibrational loads also need to be taken into account.

Aging of adhesives occurs due to hydrolysis and thermo-oxidative degradation. In most applications, the product would be exposed to both oxygen and water, and both reactions would be expected to occur simultaneously. An aging model that quantifies the aging rate as a function of temperature and humidity exposure, such as (4) for the case of OxySIM 600 SPIE adhesive, can be used to calculate acceleration factors for accelerated aging tests.

The second factor contributing to deadhesion is fatigue. Crack growth rates vary depending upon the loading, the ambient temperature and humidity, as well as the amount of aging that has occurred. If a fatigue damage model for an adhesive that predicts the crack growth rate as a function of these factors is available, such as the model provided in [25] for a particular underfill epoxy, it can be used to design the accelerated fatigue test for the product.

If no such model can be found, and sufficient time and resources are not available for a model to be developed by the user of the adhesive, an alternate approach for test design is to use time compression for applied stresses. Although less accurate than using a fatigue damage model to design the test, time compression addresses most of the factors that affect the growth rate of a fatigue crack and can serve as an approximation. The same number and magnitude of cycles that would be encountered in the actual application are used in the test, but ramp rates and frequency of the cycles are increased. To account for the dependance of crack growth rate upon the amount of aging that has occured, the aging should be spread out throughout the cycling process. For some products, this can be accomplished by instituting a large dwell time on the high end of each thermal cycle during which aging can occur. If a larger acceleration factor for aging is needed, samples could be passed back and forth several times between a high temperature, high humidity HAST chamber and a temperature/humidity cycling chamber, such that when the test was completed, both an entire design lifetime of aging damage and fatigue damage had been accumulated by the product. If bending loads, shocks, or vibrations were also expected to be encountered in the application environment, then these loadings could be applied in series or in parallel with the temperature/humidity cycling.

D. Calculation of Acceleration Factors for Aging

The goal of an accelerated aging test is to incur the same amount of damage as would be incurred in the actual application, but in a shorter period of time. The acceleration factor (AF) for an accelerated test is defined as the ratio of the time to failure at application conditions and the time to failure at test conditions, keeping the failure modes and mechanisms the same.

Tests must be designed such that overstress limits are not exceeded. For adhesives that are designed to be used below their glass transition temperature in the field, the glass transition temperature should also not be exceeded during testing. Tests should be designed with as large an AF as possible to minimize the time required for testing, and test conditions are often chosen by setting load levels just below the overstress limits. Preliminary tests can be run if needed to help in identifying these limits. Once test conditions have been decided, the duration of the test is then calculated using the AF equation.

For a process that follows first-order reaction kinetics, the AF can be calculated by considering that the percent degradation experienced in the application during the design life, t_A, at rate k_A, must be equal to the percent degradation obtained in the test in time t_T and at rate k_T. Using (4a) for the case of OxySIM 600, this can be expressed mathematically as

$$100 \cdot e^{-k_A t_A} = 100 \cdot e^{-k_T t_T} \tag{5}$$

where the reaction rates, k, are calculated using (4b). The AF for an accelerated test is then

$$AF = \frac{k_T}{k_A} \tag{6}$$

which can be used to calculate the time required for a test by dividing the design life by the AF

$$t_T = \left(\frac{k_A}{k_T}\right) t_A. \tag{7}$$

IV. Case Study: Qualification of a Naval MCM

In order to illustrate the application of the proposed qualification methodology, a test plan is derived for a multi-chip module (MCM). The MCM uses OxySIM 600 SPIE as a lamination adhesive, in which it bonds a Cu-metallized polyimide surface to a bare polyimide surface. The MCM is part of a radar system and will be operated in an electronics room aboard a naval vessel. The design life of the product is five years. In order to simplify the analysis, only the testing needed to evaluate the resistance of the modules to SPIE deadhesion will be considered. In an actual test plan, additional tests would be run in parallel to evaluate other potential failure mechanisms (i.e., electromigration of metallization).

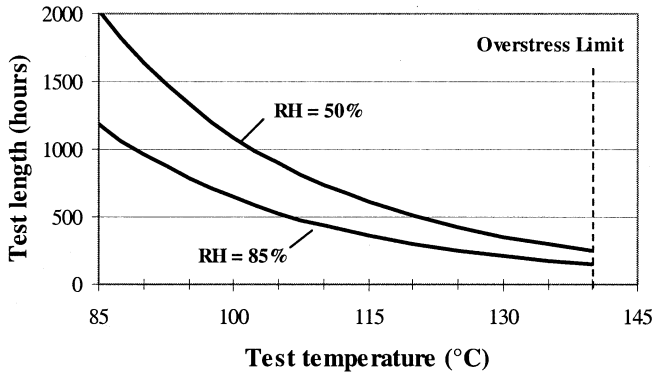

Fig. 5. Aging test length as a function of temperature and humidity settings.

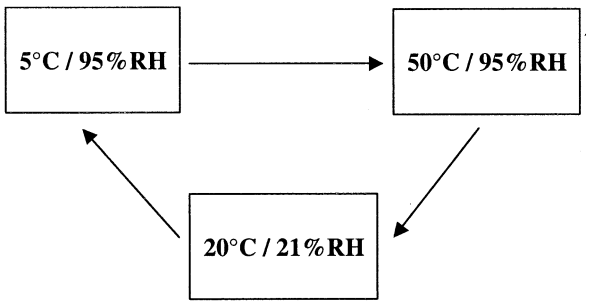

Fig. 6. Temperature/humidity cycling conditions.

A. Application Environment

The MCM being qualified will experience both temperature and humidity cycling in its local environment. It will be located in a room maintained at an average of 25 °C and 50% RH during missions. The MCM is attached to a cold-plate that removes heat due to power dissipation and maintains the device at 25 °C. At the end of each mission, which on average lasts two weeks [33], the environmental controls may be disabled while maintenance is performed. Environmental conditions during maintenance will depend upon the time of year and the location of the ship in the world, but since the device is considered "reliability-critical," worst-case conditions are used to provide a large safety factor. According to the Naval Ocean Systems Center [33], the extremes of the conditions that may be found in an electronics room under uncontrolled circumstances are 22 °C/25%RH (worst case low temperature/low humidity), 0 °C (worst case low temperature, at which RH is undefined but would be high as freezing was approached during cooling), 50 °C/21%RH (worst case high temperature/low humidity), and 30 °C/95%RH (worst case high humidity). Temperature therefore varies from 0–50 °C, while humidity varies from 21-95%.

The only mechanically applied loading to which the MCM may be exposed is shock. According to the design specification, the MCM must be able to withstand six pulses at 1500 g's with 0.5 ms durations. The polyimide will not experience any fluctuating bending loads, as it is mounted on a rigid substrate. Vibration loading will be negligible due to the manner in which the devices are mounted, and their location aboard the ship.

B. Determination of Aging Stage Test Parameters

The aging of the module can be approximated as occurring at 25 °C/50%RH for five years (43,830 h). The reaction rate for the aging of the SPIE at this application condition can be calculated using (4b) as 5.126×10^{-5} hours^{-1}.

The first step is the determination of overstress limits, as an aging temperature and humidity below the overstress limit must be chosen for use. The temperature should be kept below 150 °C to stay well below the glass transition temperature of the SPIE (approx. 165 °C), and the humidity should be kept below 95% to prevent condensation. For a given test condition, the aging time required to produce an amount of damage equivalent to the application condition can be calculated using (7). A graph of the aging times that would be required for various temperature and humidity settings is provided in Fig. 5. Since an adequate AF can not be obtained with prolonged dwells during the temperature/humidity cycle for the product, aging will need to be performed at a higher temperature in multiple stages, intermittent with the fatigue stages. If 130 °C/85%RH were chosen as the aging condition, a total of 213 h (nine days) of aging would be necessary. If three aging stages were incorporated in the test flow, each stage would then last for three days.

C. Determination of Fatigue Stage Test Parameters

Since a fatigue damage model is not available for SPIE adhesive, time compression will be used for the fatigue loading. One cycle that could be chosen to represent the application environment is to cycle between 5 °C/95%RH and 50 °C/95%RH, with a dwell at 20 °C/21%RH during temperature decent (see Fig. 6). The dwell during decent forces humidity to be lowered before temperature, which prevents condensation from forming on the product. It also captures the full damage potential of the humidity cycling of the environment on the product by ensuring as low a RH is reached on the low end of the cycle as possible. A low temperature of 5 °C was used instead of 0 °C due to test chamber limitations, and knowledge that the local environment in the room in which this product will be used will never be allowed to reach freezing conditions. Each dwell need only last long enough for the MCM to reach temperature and moisture equilibrium, which can be determined experimentally, or by thermal and diffusion modeling. Each ramp rate could be set equal to the equipment capabilities. This cycle would alternate between the two worst-case scenarios for down-time conditions, and each cycle would represent one month. The mission condition is not used directly, as this condition is transgressed during each cycle between worst-case downtimes. For a five-year design life, a total of 60 cycles would be required. If three fatigue stages are used in the test design, 20 cycles are needed at each stage.

In addition to temperature and humidity cycling, this application environment includes shock conditions. Since these shocks would be assumed to occur due to near or direct hits during combat, they can be assumed to occur while the ship is on mission, when the modules would be at 25 °C/50%RH. Following each of the three temperature/humidity cycling stages, two shocks should be applied to the modules in a 25 °C/50%RH environment.

At the end of the fatigue testing, the worst-case environment that the module could see would have been experienced. An

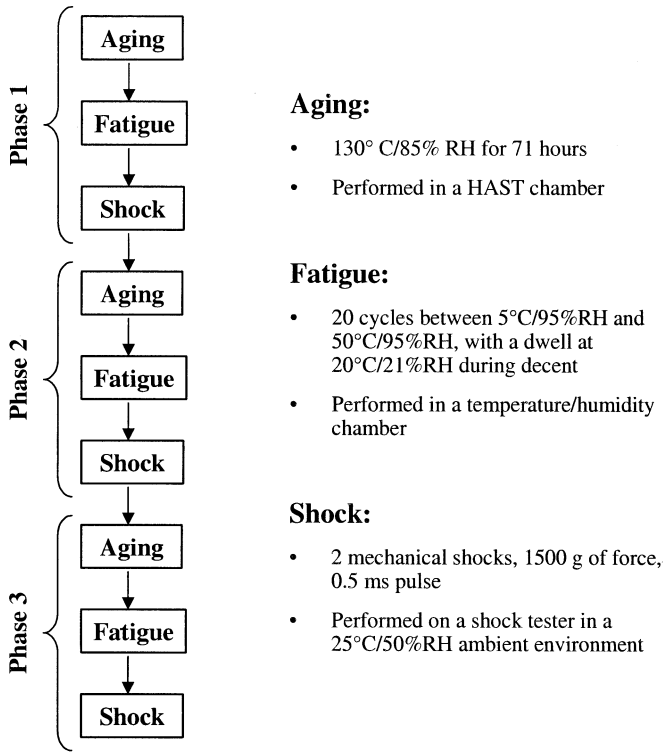

Fig. 7. Test flow for SPIE deadhesion test.

additional safety factor could be applied if desired by increasing the size or number of the cycles or shocks applied.

D. Qualification Test Flow

The resulting test flow plan to evaluate the MCM for resistance to SPIE deadhesion is illustrated in Fig. 7. This test plan produces the same amount of aging and fatigue damage that would be expected to be incurred in the worst-case application environment over the five-year design life. Following test completion, a full electrical test and acoustic microscopy would be performed. If desired, these tests could also be run following each of the three stages in order to check for damage throughout the test. If no signs of deadhesion were found following completion of the test flow, the product would be considered qualified against SPIE deadhesion for the given application environment.

V. Conclusion

An empirical model for the environmental aging of a siloxane-polyimide-epoxy adhesive has been developed. Both temperature and humidity were found to affect the rate of aging. The aging and peel test methodology used to develop this model could be used to develop aging models for other types of adhesives.

An accelerated testing methodology to evaluate the resistance of products to deadhesion that combines environmental aging with fatigue testing and captures the interactions between these two damage mechanisms has been proposed. Aging models can be used to design accelerated aging tests, which are combined with fatigue tests to qualify a product for a given application environment. This methodology overcomes some of the limitations of commonly used industry qualification techniques, and more accurately evaluates the resistance of adhesive materials to deadhesion.

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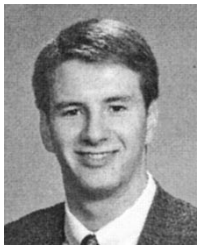
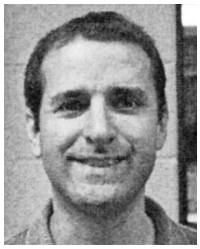


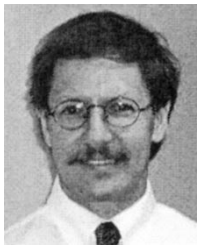
Steven Murray received the B.S. degree in mechanical engineering from the University of Wisconsin, Madison and the M.S. degree in mechanical engineering from the University of Maryland, College Park.

He is currently with Northrop Grumman Electronic Systems and works on the design of sensors and radar systems. Prior to his current position, he performed electronic packaging research at the CALCE Electronic Products and Systems Center, and worked on the design of cellular phones at Motorola.



Craig Hillman received the Ph.D. degree in materials from the University of California, Santa Barbara.

He is the Director of Laboratory Services, CALCE Electronic Products and Systems Center (EPSC), University of Maryland, College Park. He performed post-doctoral work at Cambridge University, Cambridge, U.K., before accepting his current position at CALCE EPSC. His areas of interests are concerned with identifying and characterizing failure mechanisms in electronic products and systems. Major areas of work include conductive filament formation, crack initiation in ceramic capacitors, degradation of electrolytic capacitors, and failure mechanisms in plastic encapsulants.



Michael Pecht (F'92) received the B.S. degree in acoustics, the M.S. degree in engineering, and the M.S. and Ph.D. degrees in engineering mechanics, all from the University of Wisconsin, Madison.

He is the Director of the CALCE Electronic Products and Systems Center, University of Maryland, College Park, and a Full Professor with a three-way joint appointment in mechanical engineering, engineering research, and systems research.

Dr. Pecht is an ASME Fellow. He served as Chief Editor of the IEEE TRANSACTIONS ON RELIABILITY for eight years and on the Advisory Board of IEEE SPECTRUM. He is currently the Chief Editor for *Microelectronics Reliability*, an Associate Editor for the IEEE TRANSACTIONS ON COMPONENTS AND PACKAGING TECHNOLOGIES, and on the Advisory Board of the *Journal of Electronics Manufacturing*. He serves on the Board of Advisors for various companies and consults for the U.S. government, providing expertise in strategic planning in the area of electronics products development and marketing.